IS 3926 (1986): Methyl Cinnamate [PCD 18: Natural and Synthetic Fragrance Materials]

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Indian Standard

SPECIFICATION FOR METHYL CINNAMATE

(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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AMENDMENT NO. 1 MAY 2005
TO
IS 3926 : 1986 SPECIFICATION FOR METHYL CINNAMATE

(First Revision)

[*Page* 5, *Table* 1, *Sl No.* (iv), *col* 3] — Substitute '99' *for* '98'.

(PCD 18)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR METHYL CINNAMATE

(First Revision)

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Indian Standard

SPECIFICATION FOR METHYL CINNAMATE

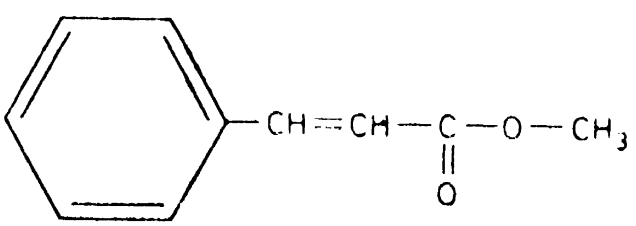
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 October 1986, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This Indian Standard was first published in 1966. The Sectional Committee responsible for its preparation felt that it should be revised with a view to bring it in line with trade practices in perfumery technology and also to align it with the quality of the material currently manufactured and sold in the country.

0.3 Methyl cinnamate ($C_{10}H_{10}O_2$) is found as the main constituent in oil of a physiological variety of *Ocimum canum* Sims., fam. Labiatae and in oil of galanga. It is a commercially available perfumery chemical having strong fixative qualities and for which property it is frequently employed in amber type colognes. It is also used as a soap perfume and for spicy and oriental blends. It helps to impart carnation and narcissus notes in perfumery. It is used in both perfumes and flavours for its spicy and berry-like characters. It has the following structural formula:



Methyl cinnamate (Molecular Mass 162·19)

0.4 A new requirement of methyl cinnamate, percent by mass, minimum along with gas chromatographic analysis for determination of methyl cinnamate has been incorporated in this revision based on data generated through indigenous testing. Requirement of relative density has also been incorporated.

0.5 In the preparation of this standard, considerable assistance has been derived from the following:

Givaudan index. 1961. Ed 2. Givaudan-Delawanna Inc, New York.

EOA No. 59-1964 Standard for methyl cinnamate. Essential Oil Association of USA, New York.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for methyl cinnamate.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 Methyl cinnamate shall be obtained by esterification of methyl alcohol with cinnamic acid.

3.1.2 The material shall be a white crystalline mass, free from extraneous matter and adulterants.

3.2 **Solubility** — The material shall be clearly soluble in 4 parts of ethyl alcohol (80 percent by volume), when tested as prescribed in IS : 326 (Part 6)-1986‡.

3.3 The material shall also be tested olfactorily and specially for the by-notes as prescribed under 4 and 5 of IS : 2284-1963§.

3.4 The material shall also comply with the requirements given in Table 1.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

‡Methods of sampling and test for natural and synthetic perfumery materials: Part 6 Determination of solubility in ethanol (*second revision*).

§Method for olfactory assessment of natural and synthetic perfumery materials.

TABLE 1 REQUIREMENTS FOR METHYL CINNAMATE

(*Clauses* 3.4 *and* 6.1)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Clause No. in Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Odour	Fruity, balsamic odour	4 and 5 of IS : 2284-1963*	—
ii)	Colour and appearance	White to yellowish crystalline solid	—	—
iii)	Acid value, *Max*	2·0	IS : 326 (Part 7)-1980†	—
iv)	Methyl cinnamate, percent by mass, *Min*	98	—	A
v)	Freedom from chlorinated compounds	To past test	16.10 of IS : 326-1968‡	—
vi)	Freezing point, °C, *Min*	33·8	IS : 326 (Part 18)-1984†	—

*Method for olfactory assessment of natural and synthetic perfumery materials.

†Methods of sampling and test for natural and synthetic perfumery materials:

Part 7 Determination of acid value (*second revision*).

Part 18 Determination of freezing point.

‡Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

4. PACKING AND MARKING

4.1 The material shall be supplied in glass bottles, tins or in any suitable container as agreed to between the purchaser and the supplier.

4.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative sample of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

5.2 Number of Tests — Tests for determination of all the characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity — The lot shall be considered as conforming to the specification if the composite sample satisfies all the requirements specified in this standard.

6. TEST METHODS

6.1 Tests shall be conducted as prescribed under **3.1, 3.2, 3.3, 3.4** and the appropriate references specified in col 4 and 5 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (iv)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF METHYL CINNAMATE

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

†Specification for water for general laboratory use (*second revision*).

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatograph for methyl cinnamate using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

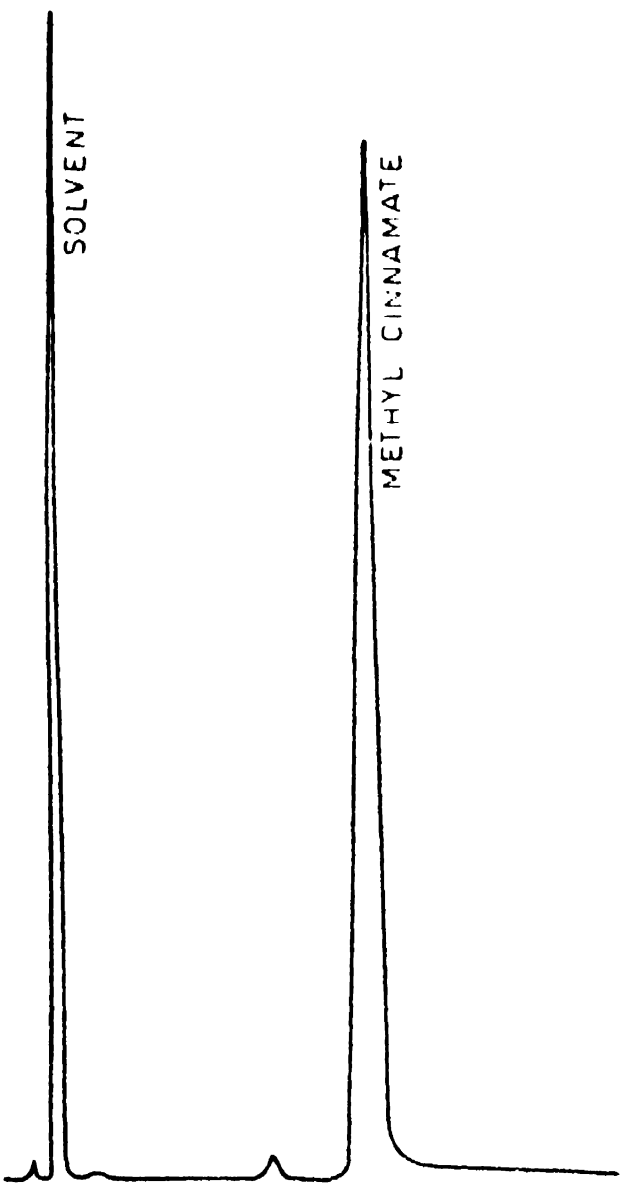


FIG. 1 TYPICAL CHROMATOGRAPH OF METHYL CINNAMATE

a) *Sample*: Methyl cinnamate

1) Material	Copper
2) Length	5·496 m
3) Outer diameter (OD)	0·635 cm
4) Inner diameter (ID)	0·476 cm
5) Stationary phase	FFAP*, 10 percent by mass
6) Solid support	Chromosorb WAW 60-80 Mesh

b) *Carrier Gas*: Hydrogen

c) *Conditions*

1) Column temperature *iso*-thermal	175°C
2) Injection port temperature	220°C
3) Carrier gas flow	40 ml/min
4) Inlet pressure	3·5 kg/cm^2

d) *Detector*

1) Type	Thermal conductivity
2) Temperature	280°C

e) *Recorder*

1) Span	1 mV
2) Chart speed	0·254 cm/min

f) *Attenuation*: 4

NOTE — This analysis may also be accomplished with columns containing carbowax — 20 M, DE. G.S (Diethylene Glycol Succinate).

A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject the sample (dissolved in the suitable solvent) at inject port where it is vaporized and well mixed with the carrier. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As different constituents pass through the detector, they give signals corresponding to the amount of particular constituents leaving the column. The detector signals, on transmission to the recorder, plot the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents.

*Free Fatty Acid Phase (FFAP) is carbowax 20 M treated with nitrophthalic acid.

A-3. CALCULATION

A-3.1 Area Measurement (*see* Note 1) — Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-3.2 Area Normalization (*see* Note 2) — By normalizing, it is meant, calculating the percentage composition, by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurement, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

(*Continued from page* 2)

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Kalai Kathir Building, 670 Avinashi Road, COIMBATORE 641018	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	71 19 98
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	320 10 84
E-52, Chitranjan Marg, C- Scheme, JAIPUR 302001	37 38 79
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